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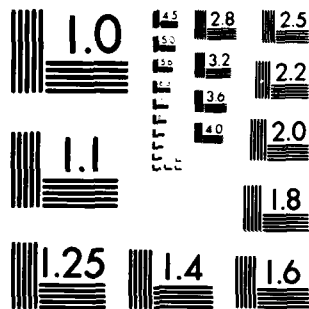
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PRESSURE DEPENDENCE OF THE GLASS TRANSITION AND
RELATED PROPERTIES OF AN ELASTOMER-SOLITHANE 113

by

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PRESSURE DEPENDENCE OF THE GLASS TRANSITION
AND RELATED PROPERTIES OF AN ELASTOMER - SOLITHANE 113

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Abstract

Several studies have been carried out on a polyurethane elastomer (Solithane 113). At atmospheric pressure dielectric methods were used to study ϵ'' over the temperature range from -180°C to $+20^{\circ}\text{C}$. Three peaks in ϵ'' were observed: an α peak associated with the glass transition and two low temperature peaks (β and γ). Mechanical tests were performed to observe the effect of the pressure induced glass transition, P_g , on the Young's modulus. The glass transition was studied as a function of pressure from 1 bar to 6.5 kilobar by observing the shift of the dielectric α -peak with pressure and also by volumetric methods. A densification of the glass and a resultant shift in the glass transition temperature could be achieved by forming the glass at high pressures (T_g) as opposed to pressuring the glass formed at 1 bar (T_g^*). It was found that $\Delta\beta/\Delta\alpha > dT_g/dP$, but $\Delta\beta/\Delta\alpha \approx dT_g^*/dP$. At high pressures dT_g/dP reached a limiting value of 10.4°C/kb .

I. INTRODUCTION

The research studies investigated the glass transition behavior of a polyurethane elastomer (Solithane 113) and other related properties as a function of pressure and temperature. Two methods have been employed in determining the glass transition temperature: dielectric and volumetric measurements. In addition, characterization of two low temperature transitions in the elastomer has also been made.

The study of the glass transition as a function of pressure has had its share of controversy and confusion, and often theoretical predictions have gotten ahead of clear experimental results. The small amount of experimental data has been unable to establish any one of several theories as clearly superior to the others.^{1,2,3} Conclusions based on experimental results have been varied owing to the variety of ways in which the glass can be formed in pressure-volume-temperature space and also due to the difficulty of measuring certain quantities accurately, especially the change in compressibility across the transition. The kinetic nature of the observed glass transition also complicates any thermodynamic understanding of the phenomenon.

Free volume theories are based on the assumption that the fractional free volume, $f = f_g + \alpha_f(T - T_{g0}) - \beta_f(P)$ is constant at the glass transition. Here f_g is the fractional free volume at T_{g0} , the glass transition temperature at atmospheric pressure, α_f and β_f are the thermal expansivity and compressibility of the free volume, respectively. Since the assumption is made that $f = f_g$ at the glass transition, then the free volume theory predicts a linear increase in

T_g with pressure according to $dT_g/dP = \beta_f/\alpha_f$.¹ The free volume thermal expansivity, α_f , was originally determined by fitting data from relaxation studies to the WLF equation. This leads to a "universal" value of α_f which was generally about equal to $\Delta\alpha = \alpha_L - \alpha_g$ obtained from volumetric studies. The association of β_f with $\Delta\beta$, however, has not been as successful.⁴ The discrepancies between measured values of $\Delta\beta/\Delta\alpha$ and dT_g/dP imply that free volume is not the single controlling parameter of the glass transition.^{3,5} Some have allowed for changes in free volume at the glass transition, but this is equivalent to saying that free volume is not the single ordering parameter governing the glass transition.⁶

Thermodynamic theories are based on the assumption that the glass transition is or reflects a true second order thermodynamic transition. The theory of DiMarzio, et al. obtains the transition temperature, T_2 , as a function of pressure where T_2 is the temperature at which the configurational entropy becomes zero. This theory is based on equilibrium thermodynamics; therefore it predicts the Ehrenfest relations to hold (i.e. $dT_g/dP = \Delta\beta/\Delta\alpha = TV \Delta\alpha/\Delta C_p$). The two parameters controlling the glass transition in this model are n_0 , the number of holes in the lattice approximation of the polymer, and f , the number of bonds in the flexed or high energy conformation. The pressure is assumed not to affect f , and therefore as the pressure increases n_0 decreases causing dT_g/dP to decrease. At high enough pressures n_0 becomes zero, and further increases in pressure will not increase T_g . So the theory predicts a levelling off of the T_g versus pressure curve as pressure is increased, and the authors claim this

effect will occur in the 10 Kb range.²

Experimental observations that $\Delta B/\Delta \alpha$ is usually greater than dT_g/dP whereas $TV\Delta\alpha/\Delta C_p$ is usually about equal to dT_g/dP has lead to the view that the glass transition is controlled by the excess entropy as opposed to the free volume.⁷ The value of dT_g/dP , however, can be made to equal $\Delta B/\Delta \alpha$ by measuring dT_g/dP for glasses which are formed at only one pressure.^{3,8} This has been associated with the freezing in of an ordering parameter at one glass formation pressure which would vary if the glass was formed at different pressures.³

Dielectric measurements have been used to determine dT_g/dP for a number of polymers.¹² Values of dT_g/dP measured by dielectric and volumetric methods have generally compared well.^{9,10}

In this investigation we studied the glass transition as a function of pressure by dielectric and volumetric means. The results are compared with certain thermodynamic quantities for glasses formed by different methods. We also measured the Young's modulus below and above the glass transition pressure up to 7 Kb, and the specific volume as a function of pressure from which the bulk modulus can be calculated.

II. MATERIAL

The material used is Solithane 113 which is a polyurethane based elastomer produced by the Thiokol Chemical Corporation. It is formed by the reaction of a resin and catalyst. The resin is a tri-functional isocyanate formed from castor oil and tolylene diisocyanate. The catalyst consists essentially of castor oil. By combining different ratios of resin and catalyst the degree of cross-linking

can be changed. In this study we deal with the equivoluminal composition or a 50/50 volume ratio of resin and catalyst. For this composition the molecular weight between crosslinks is approximately 2000 g/mole.⁹ The specific gravity is determined to be about 0.97 cm³/gr.

III. EXPERIMENTAL

A. Dielectric Measurements

A thin sample (~.05 cm thick), electroded on both sides, was used for the dielectric measurements. This sample was placed between two brass electrodes which in turn were placed between ceramic heating plates in order to assure uniformity of temperature. The apparatus is shown schematically in Fig. 1. Temperature was measured by a chromel-alumel thermocouple embedded in the brass electrode and the temperature of the test was varied at a rate of 1° C/min. A Piezotron-U (Toyo Seiki, Tokyo, Japan) was used to measure the dielectric constants the values of which were recorded continuously on a chart recorder. All measurements were made at a test frequency of 3 Hz.

B. PVT Measurement

A schematic drawing of the PVT apparatus is shown in Fig. 2. It consists essentially of a sample which is fixed on one end and free floating on the other end. The free end is connected to a small steel rod which rides in a linear variable differential transformer (LVDT) enabling changes in length to be measured electrically. The sample is surrounded by a cylindrical heater, and temperature is measured by a chromel-alumel thermocouple placed at the sample sur-

face. The entire apparatus was placed inside a pressure vessel filled with silicon oil as the pressure medium.

C. Method of Glass Formation and T_g Measurement

In order to study how glass formation pressure affects glass transition behavior, the tests were run in two different manners. First, the sample was cooled at atmospheric pressure below the T_g, forming the normal glass (NG). The pressure was then applied to the normal glass to the desired intensity of pressure after which the temperature was raised isobarically to record specific volume versus temperature through the glass transition region. In the second experiment, the sample was cooled from above the T_g which was achieved in the first experiment holding pressure constant, again recording specific volume versus temperature. This second method formed the equilibrium or pseudo equilibrium glass. The glass formed by lowering temperature at elevated pressures as in the second experiment is designated Tp-glass. Normal glass (NG) is a Tp-glass in which P is atmospheric pressure. Both glasses were studied in the dielectric tests.

D. Measurement of Young's Modulus

The apparatus for performing mechanical tests under high pressure is shown schematically in Fig. 3. The sample is mounted in a pressure vessel and is loaded by a hydraulically driven piston. The loading piston is mounted with strain gages to measure the applied load, while sample deformation is measured by a linear variable differential transformer (LVDT). The electrical signals from the strain gages and the LVDT are amplified and displayed on an X-Y recorder. By

use of special sample yokes both tension and compression tests can be performed, however in this study only compressive measurements were made. The Young's modulus has been identified with the initial slope of the compressive stress-strain curve.

IV. RESULTS

A. Dielectric Measurements

Fig. 4 shows the dielectric loss constant, ϵ'' , versus temperature at atmospheric pressure over the temperature range from -180°C to $+20^{\circ}\text{C}$. Three peaks in ϵ'' are observed. At 3 Hz the α -peak occurs at $+2^{\circ}\text{C}$. This peak which is by far the most prominent of the three peaks is associated with the glass transition. At about -80°C , a small β -peak occurs, which has been related to the motion of small molecules, especially water, contained in the elastomer network,¹⁰ and at about -160°C a very broad γ -peak occurs which may be associated with a local mode transition occurring in the glassy state.¹⁰

The application of hydrostatic pressure causes the α -peak to be shifted to higher temperatures. By choosing the maximum point in the α -peak to locate the transition, a measure of the pressure dependence of the glass transition temperature can be obtained. This behavior is shown in the upper solid curve in Fig. 5. The maximum in the α -peak shifts about 80°C with the application of a pressure of 6.5 kilobar. The rate of increase is constantly decreasing up to a pressure of about 2.5 kilobar after which the transition temperature increases at a constant rate of $10.4^{\circ}\text{C}/\text{kb}$. The α -peak decreases in height and broadens slightly due to the application of pressure, but this effect is small, so the error with which the maximum point can be determined is essentially unaffected by pressure over the entire pressure range.

The pressure dependence of the α -peak is not affected by how the glass was formed. In other words, the same location in temperature of the α -peak at a given pressure, is obtained when either the normal glass (NG) or Tp-glass is used for the test.

B. Volumetric Measurements

Specific volume of the normal glass (NG) and the Tp-glass was measured as described in the Experimental section with increasing temperature at various pressures up to 6 Kbar as shown in Fig. 6. The glass transition temperature was then located by the intersection of straight lines drawn through the points below and above the T_g . The upper curves labelled NG in the glassy region for tests conducted at 1 and 2 Kilobar correspond to heating the normal glass (NG). All other curves correspond to cooling from the liquid state through T_g at various pressures. The glass transition temperature determined for the NG glass will be referred to as T_g^* while the glass transition for Tp-glass will be called T_g .

The normal glass has a structure which is found to be less dense at subsequently applied higher pressures than that of Tp-glass at the same pressure. Consequently its specific volume is higher as shown at 1 and 2 Kilobars in Fig. 6. The intersection of these glass curves with the liquid curve will therefore be located at higher temperatures than the intersection of the glass curve for Tp-glass. This produces a T_g^* which is greater than T_g at all pressures except atmospheric pressure where the glasses were formed identically. The slight flattening of the normal glass curves near the transition indicates volumetric relaxation as T_g^* is approached from below. This

is an indication of the kinetic nature of the observed glass transition.

The thermal expansivities of the glass and the liquid, α_g and α_l , respectively, measured from the slopes of the curves in Fig. 6 both decrease with increasing pressure. The value of α_l , however, decreases much more rapidly than α_g making determination of the glass transition more difficult as the pressure is raised.

Plots of T_g and T_g^* versus pressure are shown in Fig. 5. The lower solid curve is T_g (for Tp-glass), and the dashed curve is T_g^* (for NG). As noted earlier T_g^* has a higher value and increases more rapidly than T_g . The curve for T_g versus pressure is observed to parallel the curve obtained from dielectric measurements quite well. The volumetric glass transition being at all pressures about 20°C below the dielectric curve obtained with the frequency 3 Hz.

The slopes of the curves in Fig. 5 give values of dT_g/dP (or dT_g^*/dP) versus pressure. The slope of the dielectric and T_g curves are essentially the same. The values of dT_g/dP obtained in this manner can be compared with $\Delta\beta/\Delta\alpha$. Values of $\Delta\alpha$ are obtained by taking the difference in thermal expansivities above and below the glass transition: $\Delta\alpha = \alpha_l - \alpha_g$. To obtain $\Delta\beta$, the change in compressibility across the transition, the following method was used.¹¹ At a certain test pressure, as the temperature is lowered through the T_g , the pressure is perturbed slightly to measure $\beta = 1/V(\partial V/\partial P)_T \approx (\Delta V/V\Delta P)/\Delta P$. This value of β goes through a step decrease as the temperature is lowered through the transition region. From this step $\Delta\beta$ is obtained.

A plot of dT_g/dP , dT_g^*/dP and $\Delta B/\Delta\alpha$ are shown in Fig. 7. The values of dT_g^*/dP are seen to be nearly twice the values of dT_g/dP , and the $\Delta B/\Delta\alpha$'s are approximately equal to dT_g^*/dP within experimental errors. Similar results have been observed by other authors on uncrosslinked systems.^{3,8}

C. Comparison of Young's Modulus and Bulk Modulus

A pressure induced glass transition (or the glass transition pressure, P_g) occurs at room temperature within the seven Kilobar range as shown in Fig. 8. In this figure the Young's Modulus is seen to undergo an increase of more than two orders of magnitude in the 2 - 3 Kilobar range. Prior to the transition the modulus is typical of a rubber: $\sim 2 \times 10^7$ dyne/cm², and after the transition it is typical of a glass: $\sim 10^{10}$ dyne/cm². From a plot of specific volume versus pressure as shown in Fig. 9 the bulk modulus, B , can also be obtained. The bulk modulus is taken as the inverse of the slope of the curve in Fig. (9). B increases from $\sim 2 \times 10^{10}$ dynes/cm² to $\sim 8 \times 10^{10}$ dynes/cm² with the application of seven kilobars of pressure, but no abrupt change occurs at the glass transition as was the case for the Young's Modulus.

V. DISCUSSION

When a polymer is cooled through its glass transition, relaxation times of segments in the polymer go from very short to very long. There is actually a very broad distribution of relaxation times which is reflected in the breadth of the α -peak in Fig. 4. Although the peak maximum occurs at $+2^\circ\text{C}$, there are relaxations still occurring

well below -20°C . The maximum point in the α -peak corresponds to the condition where the average relaxation time equals the inverse of the test frequency, in this case 3 Hz. For this reason the maximum point in the α -peak must be in the liquid state (i.e. where large scale motions of the chain are possible). The fact that the maximum in the α -peak does not depend upon glass-formation conditions (see III-C) may be attributed to the fact that, although different glassy states might have been frozen in below T_g , by the time the temperature of the maximum in the α -peak is reached, the different initial structures will have relaxed to the same structure. Thus the frequency of test (3 Hz) becomes the overriding factor in determining the location of the α -peak.

The observation that dT_g/dP is the same for the dielectric test and for the volumetric test cooled from above T_g may be understood as follows. On cooling the sample from above T_g at a given pressure a pseudo equilibrium structure is frozen in. The glass has assumed its pseudo equilibrium structure for the particular pressure and rate of test, just as it will assume its pseudo equilibrium structure in the dielectric test causing the α -peak to depend on frequency. The normal glass is by no means an equilibrium structure, so the dielectric test and the volumetric test cooled through T_g reflect the same pseudo equilibrium transition.

Although no published data has actually shown a levelling off of the T_g versus pressure curve (i.e. $dT_g/dP = 0$) at high pressures, the curvature in some published T_g versus P data has made it appear that eventually it might level off. As mentioned earlier, this level-

ling off would be in agreement with certain thermodynamic theories of the glass transition. The data in Fig. 5 and Fig. 7, however, show dT_g/dP reaches a constant non-zero value. Although nothing can be said about the dT_g/dP behavior at higher pressures, it does not appear that it will become zero in the 10 Kilobar range as has been predicted.

As mentioned earlier and as shown in Fig. 7, $\Delta B/\Delta \alpha$ is approximately equal to dT_g^*/dP as has been observed by others as well. ΔB is usually obtained by measuring compressibilities between isobars. It can be seen, however, that the perturbation method of measuring compressibility is equivalent to measuring compressibility between isobars for the normal glass. It is, however, important to point out that $dT_g^*/dP > dT_g/dP$ which is also implied from the densification shown in Fig. 6. This means that a single glass surface is not sufficient to describe the PVT behavior of the elastomer.

The Young's Modulus has been shown to undergo a large increase as the pressure is raised through the P_g , whereas the bulk modulus shows very little change. This implies that it is the deviatoric component of the stress which reflects the abrupt change at the glass transition and not the hydrostatic component. From elasticity theory $E = 9B/(1 + 3B/G)$, and according to Fig. 8 the abrupt change in E is due only to an abrupt change in the shear modulus, G . The calculated values of G at various pressures are listed in Table 1 along with the values of E and B .

VI. CONCLUSIONS

Study of the glass transition behavior of Solithane 113 by dielectric and volumetric methods has produced several interesting results. The transition temperature by dielectric measurements did not change with glass formation conditions and over the pressure range studied the dT_g/dP values for dielectric measurements were the same as those for the volumetric method for the glass cooled from above T_g at various pressures.

The value of dT_g/dP at high pressures (up to 6.5 Kilobar) did not approach zero but instead reached a limiting value of 10.4°C/Kb . The fact that $dT_g^*/dP > dT_g/dP$ indicates that, although there is a single equilibrium liquid surface in PVT space, there are many glass surfaces depending on the method of glass formation.

Acknowledgment

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FIGURE CAPTIONS

Figure 1 - Dielectric Apparatus

Figure 2 - PVT Apparatus

Figure 3 - Mechanical Testing Apparatus

Figure 4 - ϵ'' vs. Temperature at 1 bar

Figure 5 - T_g vs. Pressure

Figure 6 - Specific Volume vs. Temperature at Various Pressures

Figure 7 - dT_g/dP , dT_g^*/dP and $\Delta S/\Delta\alpha$ vs. Pressure

Figure 8 - Young's Modulus and Bulk Modulus vs. Pressure

Figure 9 - Specific Volume vs. Pressure at 25°C

TABLE I

Bulk, Young's and Shear Moduli Versus Pressure

<u>P(kb)</u>	<u>B(x10⁻¹⁰ dyne/cm²)</u>	<u>E(x10⁻⁷ dyne/cm²)</u>	<u>G(x10⁻⁷ dyne/cm²)</u>
0	2.19	2.21	0.74
1.10	3.16	2.63	0.88
2.13	4.27	3.33	1.11
2.38	4.59	6.25	2.08
2.57	4.81	24.3	8.10
2.67	4.97	46.7	15.6
3.11	5.52	175.	58.5
3.67	6.03	403.	135.
4.11	6.35	516.	174.
5.00	6.71	728.	246.
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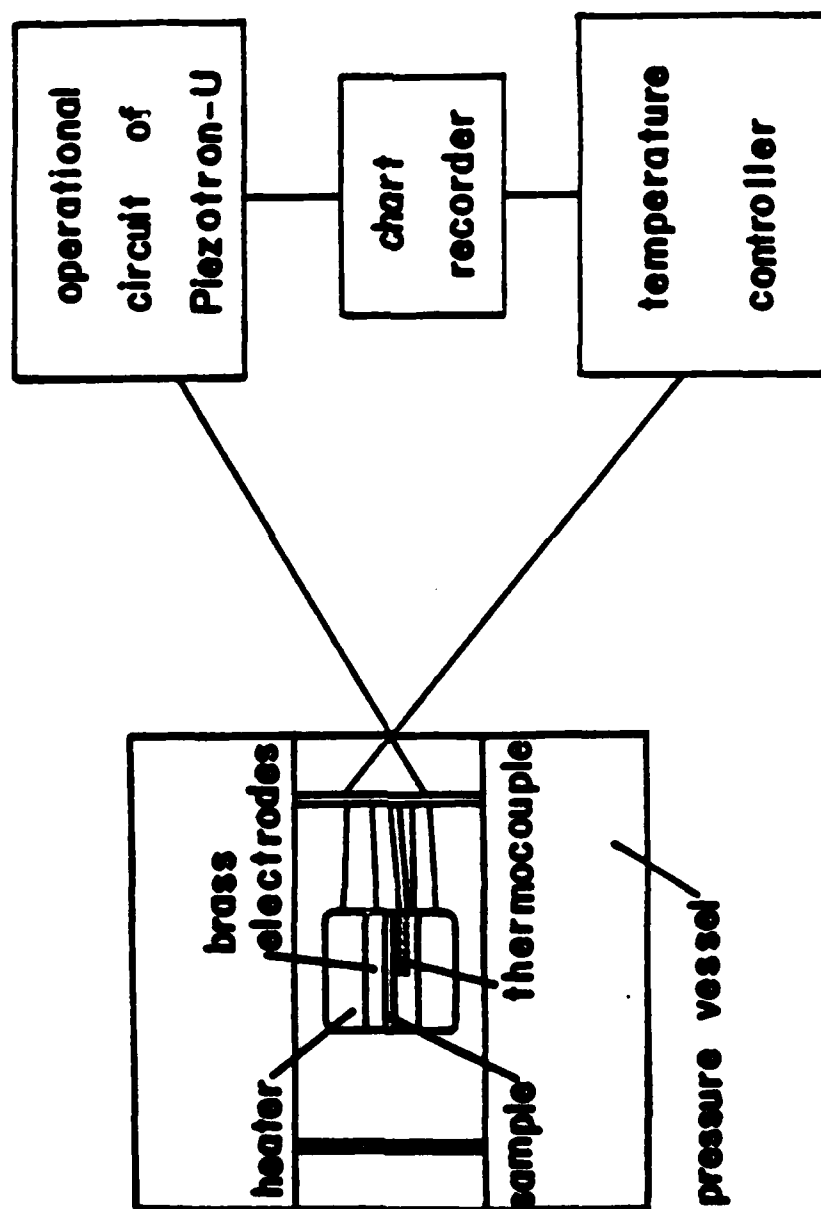


Figure 1

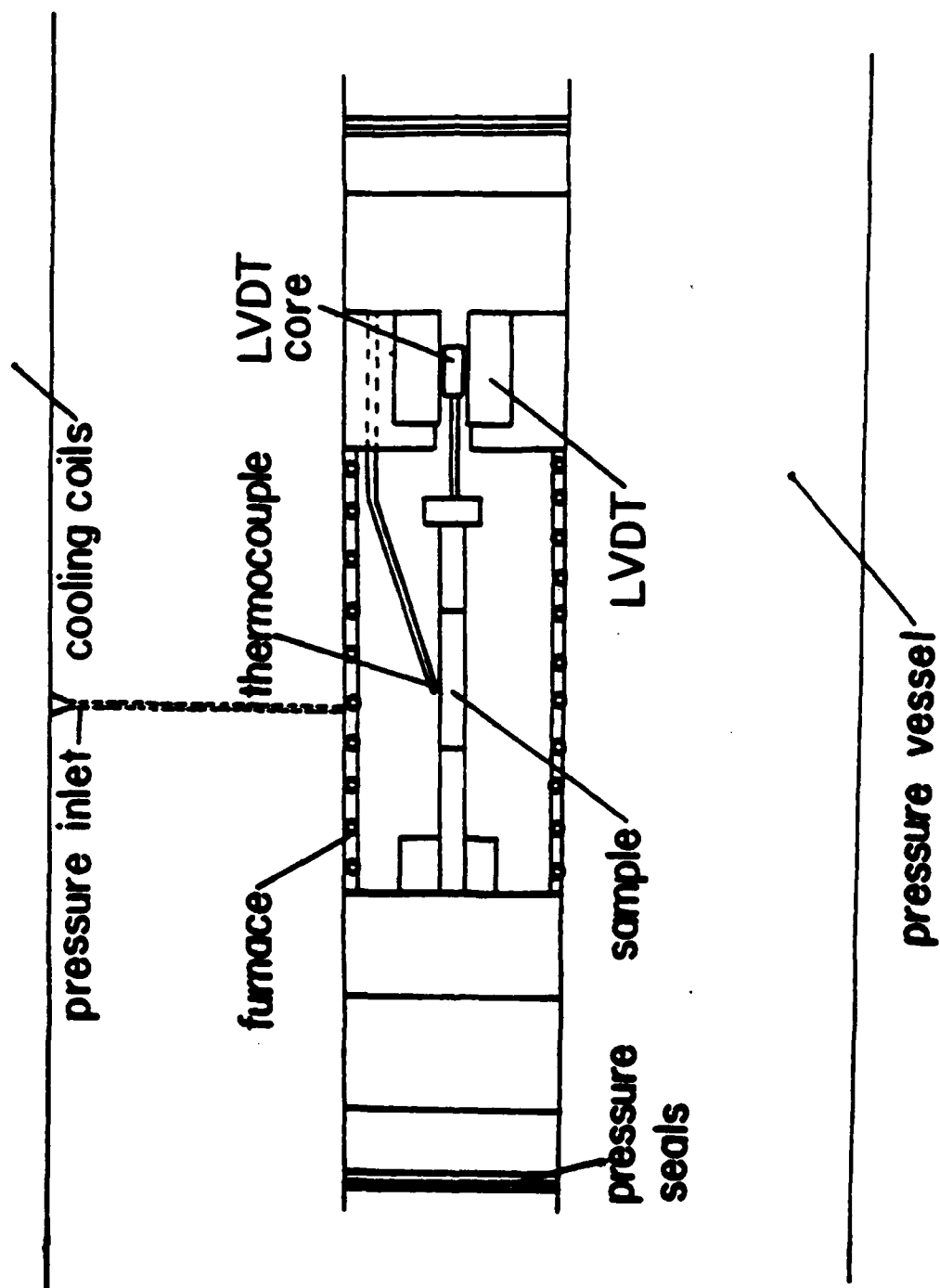


Figure 2

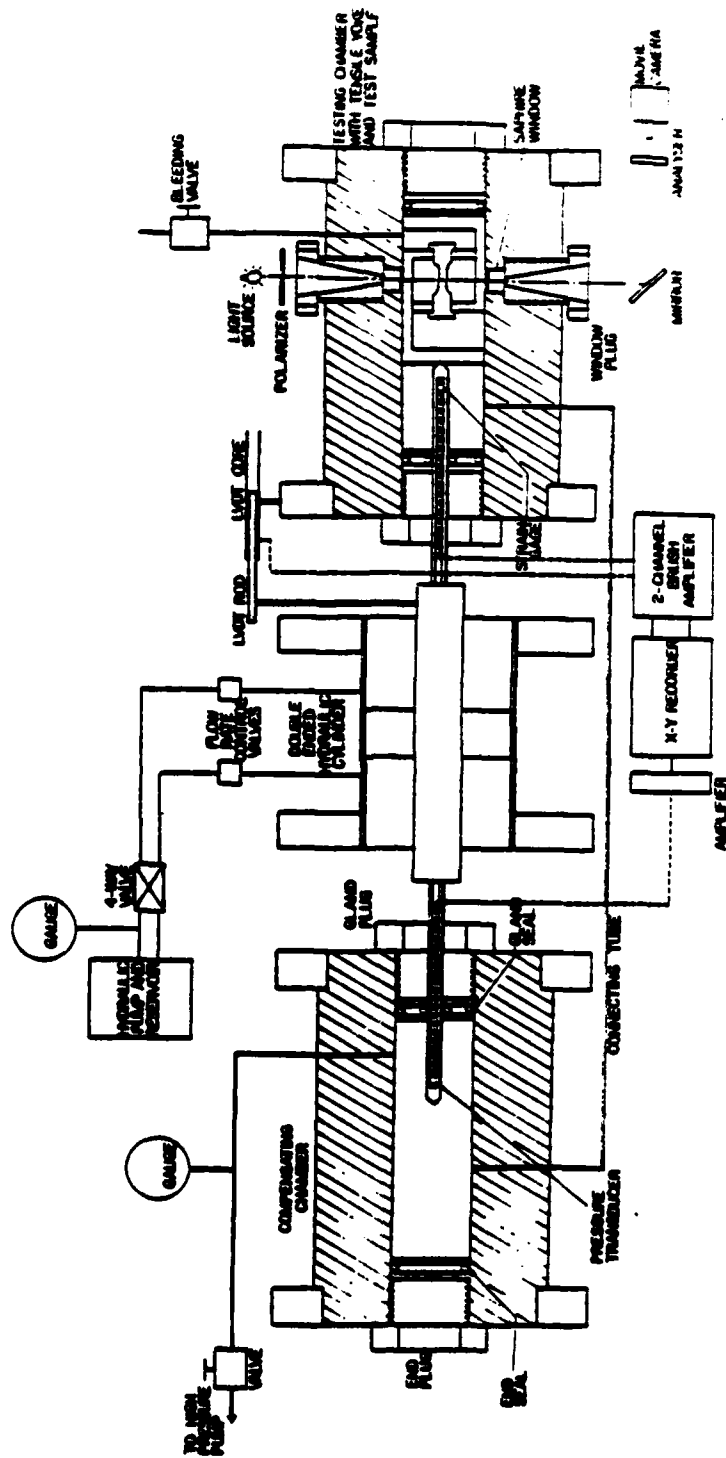


Figure 3

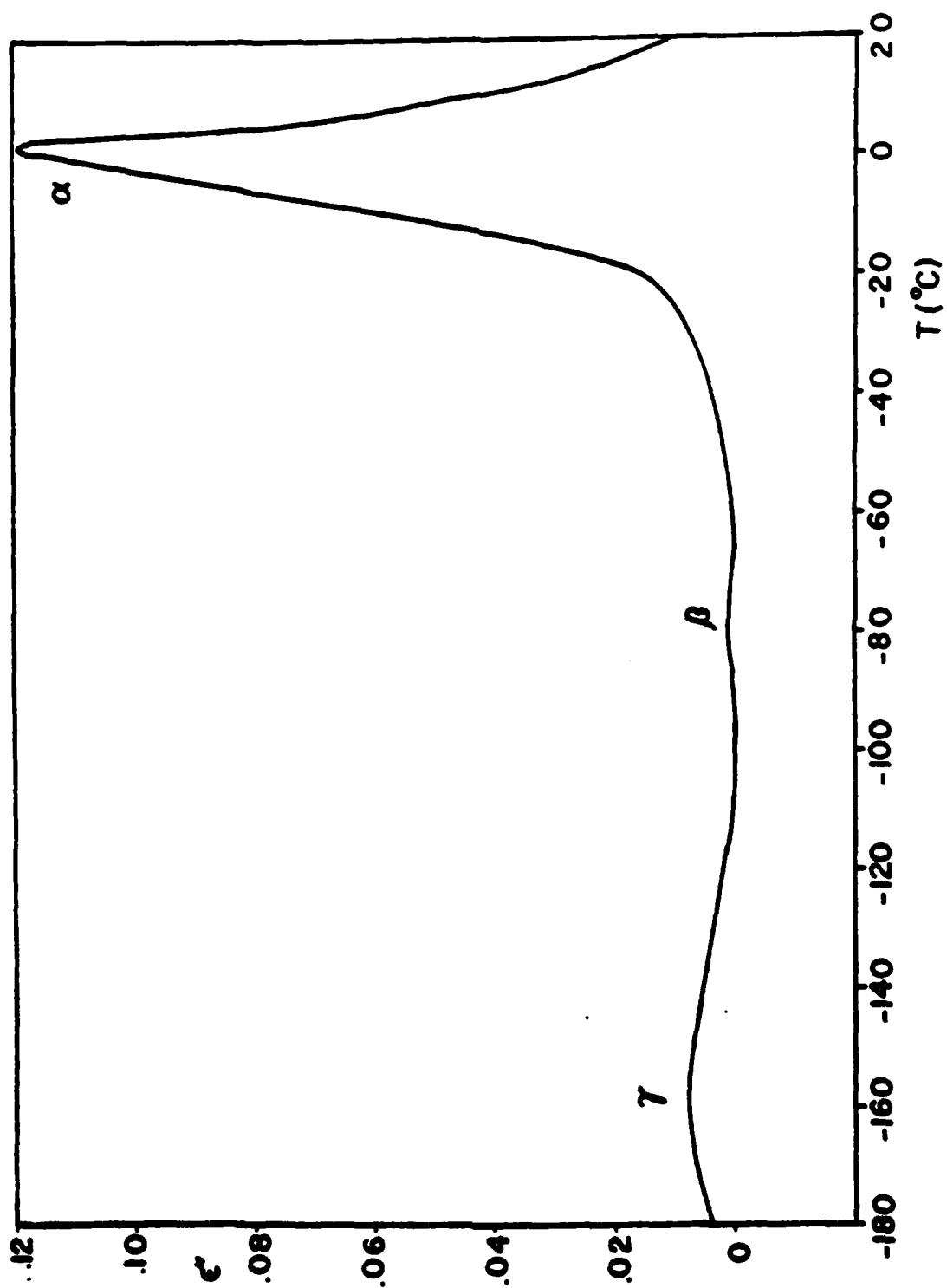


Figure 4

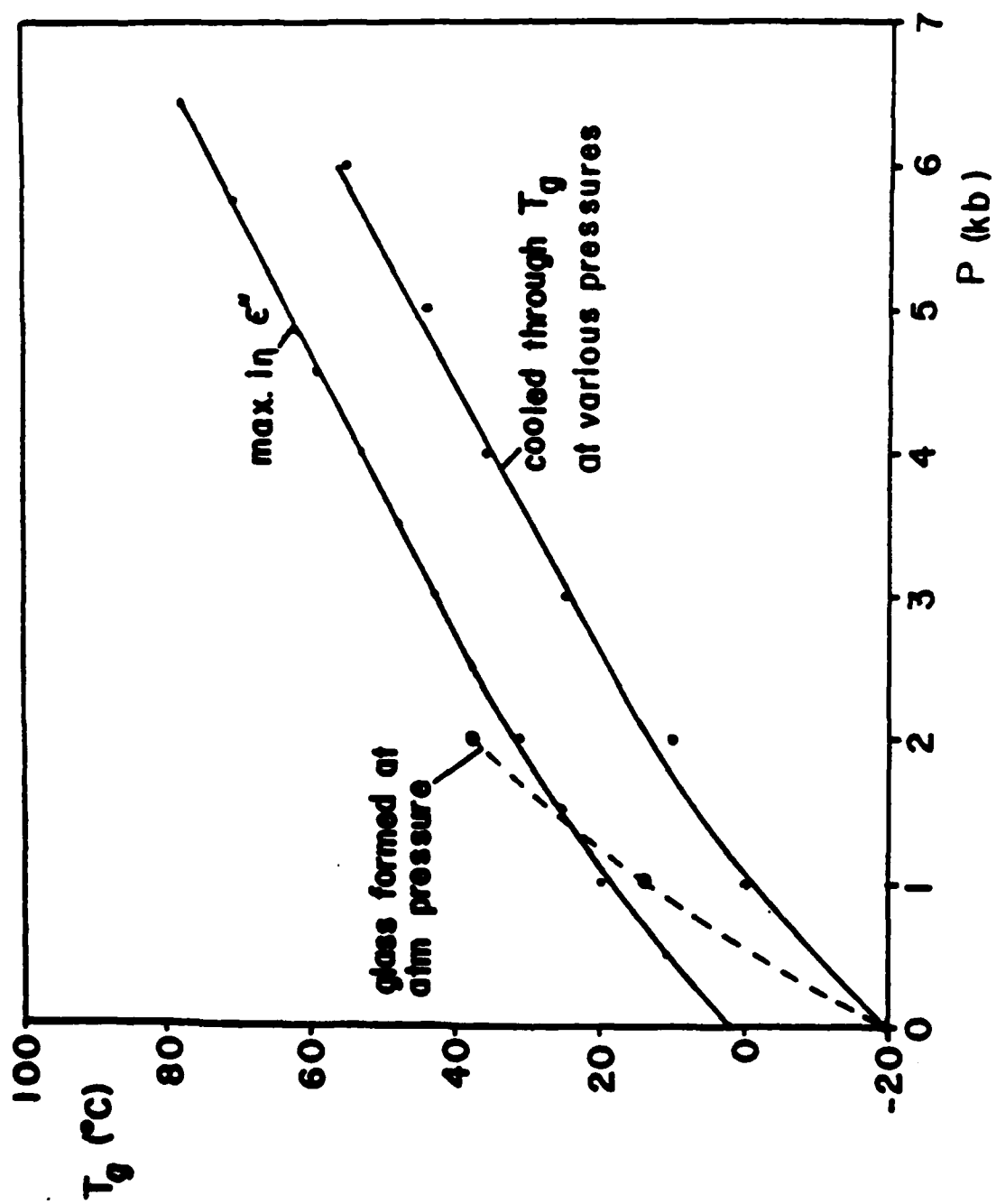


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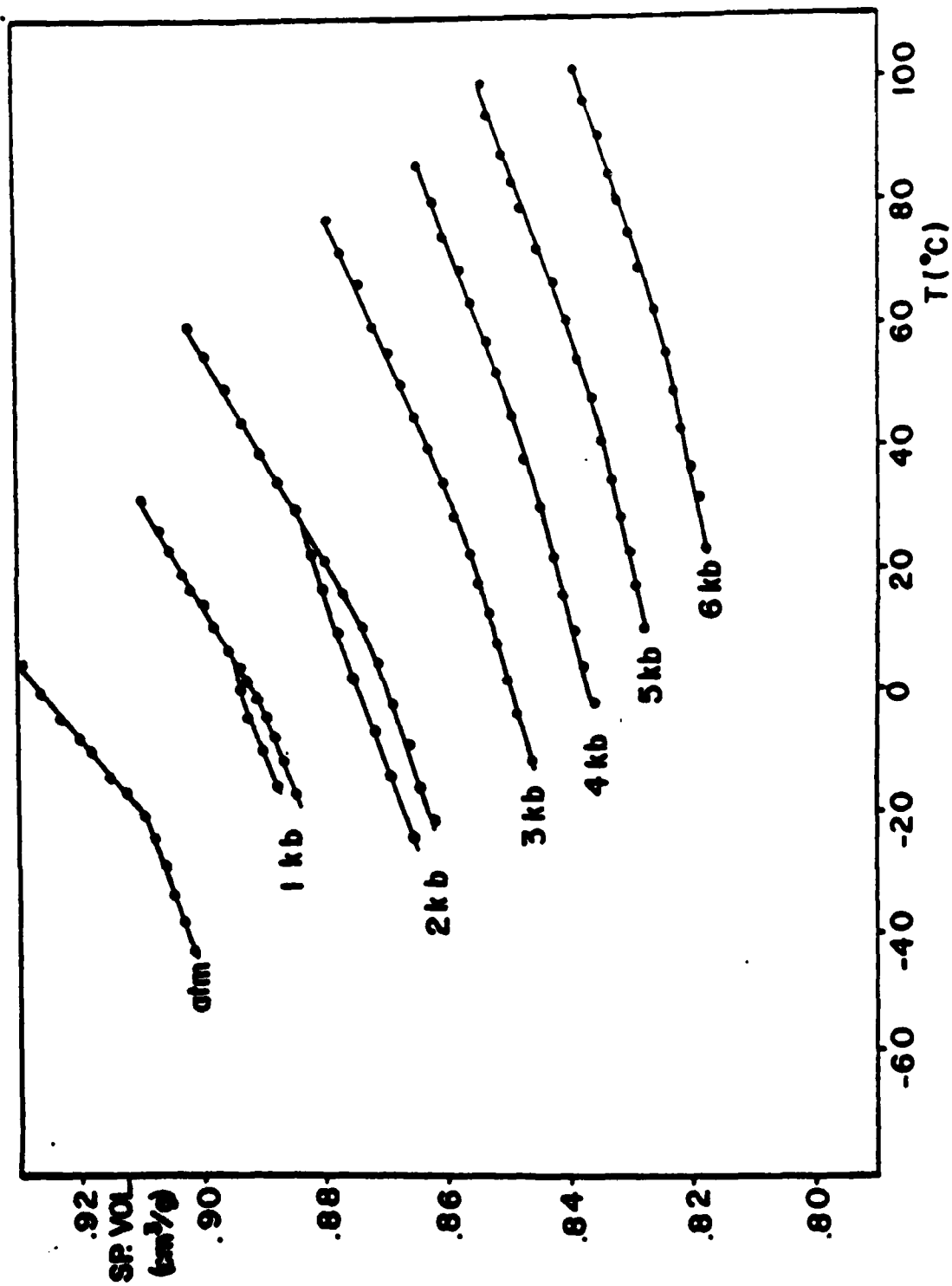


Figure 6

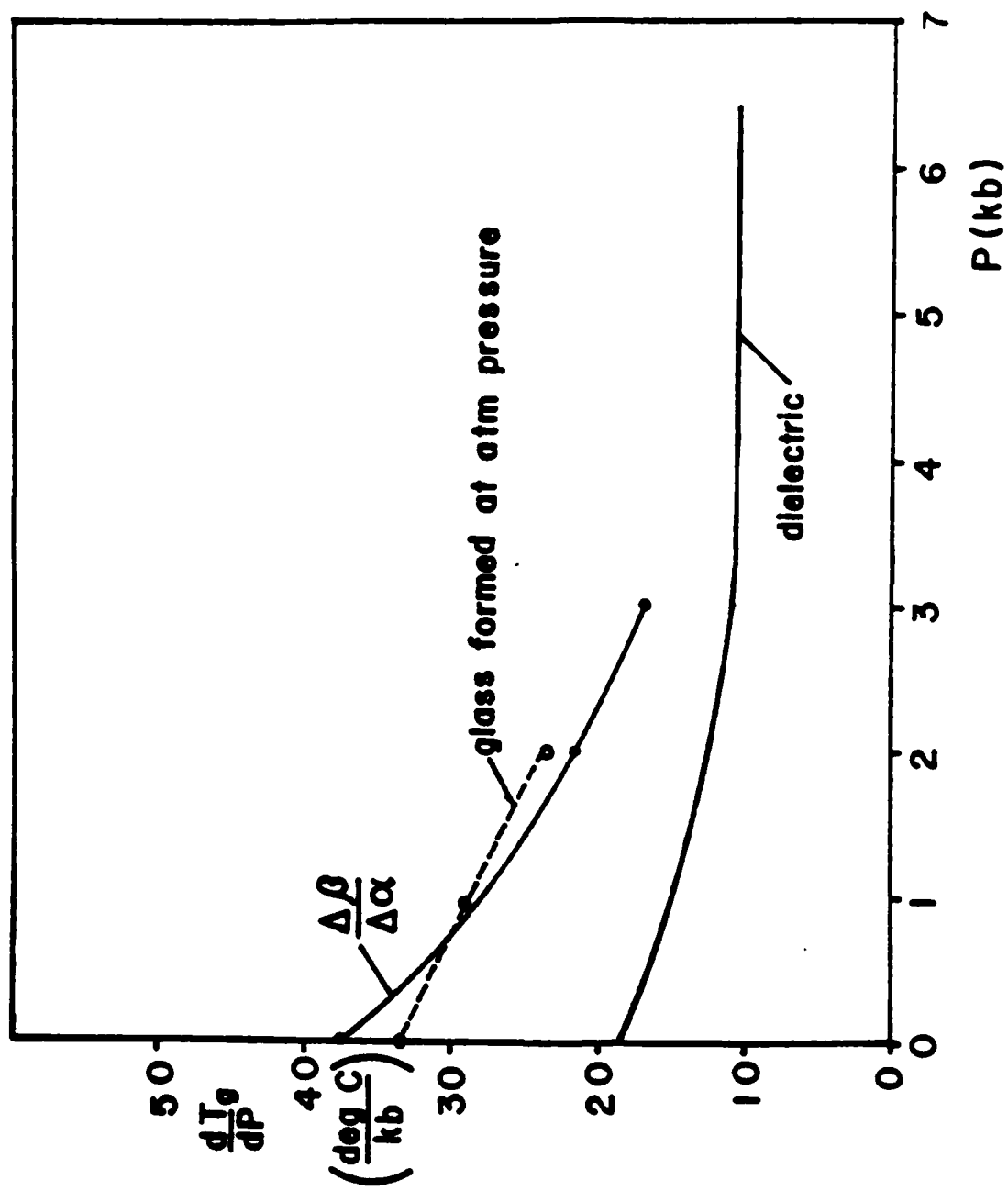


Figure 7

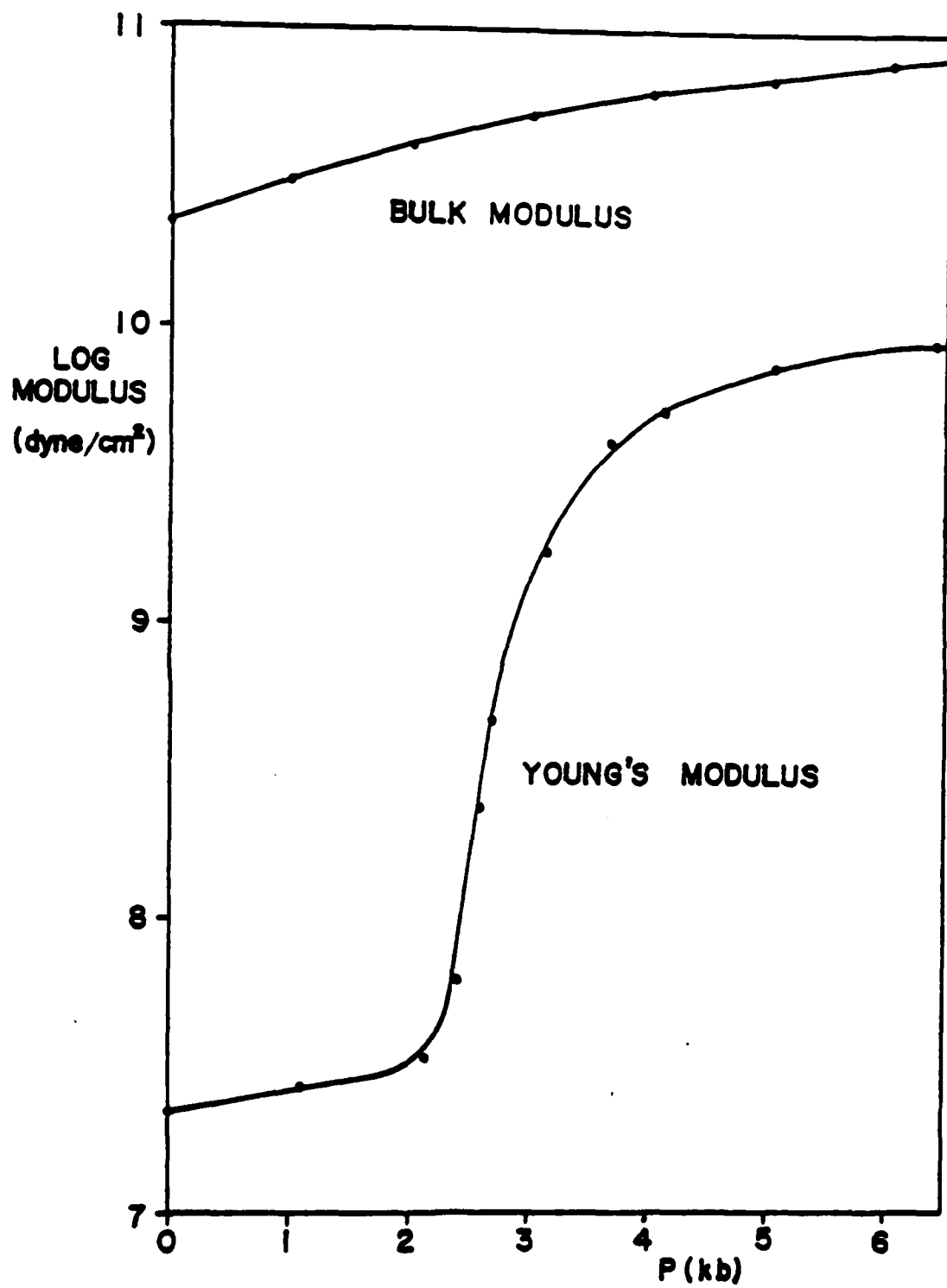


Figure 8

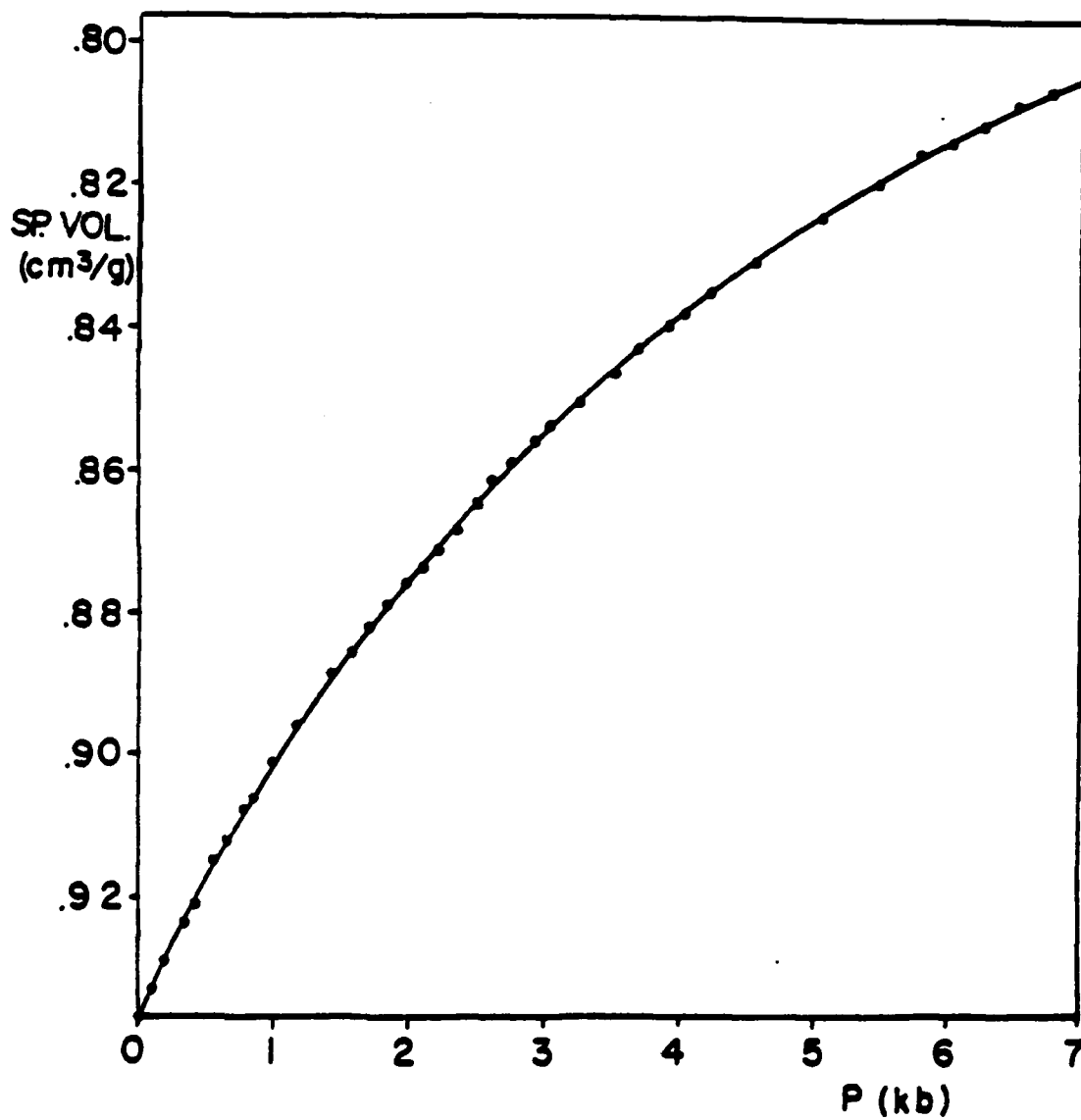


Figure 9